

## **Final Technical Report**

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### **TEM analysis of defects in 3C-SiC layers on 4H-SiC mesas**

Films discussed in this report were deposited at NASA Glenn Research Center on on-orientation 4H-SiC substrates. Substrates were patterned in order to form mesa structures of different shapes and sizes. The nominal thickness of the 3C-SiC film was approximately 10 microns although the thickness could change considerably between defect-free and defected mesa structures.

The primary objective was to identify defect structures in defect-free mesas and in particular determine the degree and mode of strain relaxation in 3C films with thickness exceeding critical thickness.

### **Sample preparation procedures**

The optical map of the entire sample surface was assembled using Nomarski contrast microscope. The defect-free mesas were identified and several 1x3 mm bars were cut using a diamond saw. The surface maps of each samples were collected again followed by gluing a sample and a sacrificial piece of silicon carbide face to face with an epoxy.



Fig. 1 Plan view optical map of the of TEM sample. Horizontal line indicates the position of the electron transparent cross section. The defect-free mesa is pointed out by an arrow.

The samples were thinned down by mechanical polishing to approximately 100 microns followed by ion beam milling. The surface morphology of the cross-sections was monitored in order to assure the exact location of the thin sections. The image of TEM sample prepared from the piece in Fig. 1 is shown in Fig. 2.

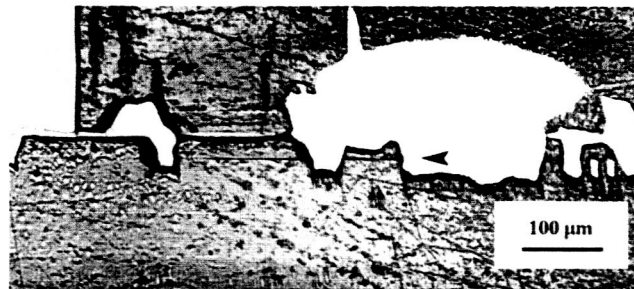


Fig. 2 Optical image of the TEM cross-sectional sample prepared at the location shown in Fig. 1. The defect-free mesa is pointed out by an arrow.

The cross section of area visible in Fig. 1 is facing upward with the defect-free mesa pointed out by an arrow. The two of the large hexagonal mesas on the left side of a defect-free one in Fig. 1 are visible on the left side of the cross-section. The 3C epilayer is visible in the cross section due to the change of color between polytypes. The average thickness estimated on several mesas is approximately 20 microns.

## Transmission Electron Microscopy

### Stacking faults

NASA Glenn team developed a technique of deposition of defect-free 3C-SiC epilayers on 4H-SiC substrates. The technique relies on growth on patterned substrates with mesa structures with sizes between 50 and 500  $\mu\text{m} \times \mu\text{m}$  defined by reactive ion etching. Some of the mesa surfaces are intersected by screw dislocations with the Burgers vector  $1c[0001]$  and the line direction approximately along the c-axis of the crystal, while others do not. Screw dislocations are well known to serve as the step sources in SiC growth on basal plane seeds and serve as “polytype-memory” defects. Their Burgers vector is a multiple of the c-lattice parameter and steps generated by screws replicate the polytype stacking sequence. Therefore, it was expected and later proven experimentally that growth on screw-containing mesas is of 4H polytype.

The polytype of the deposit on screw-free mesas can be determined by two factors. One is the pre-existing atomic steps due to intentional or non-intentional miscut of the substrate. If the step density is high (as on intentionally off-orientation substrates), then the growth proceeds by the step-flow mechanism repeating the stacking sequence of the steps. For high super-saturations and/or low miscut angles, the adatom density on the terraces is relatively higher than on the miscut substrate and the likelihood of 2D nucleus formation is increased as well. The stacking sequence in SiC produced by two dimensional nucleation on the basal plane template should correspond to the thermodynamically stable polytype. It is widely assumed that at typical CVD growth conditions (growth temperature  $<1650^\circ\text{C}$ , carbon-rich growth ambient), the stable polytype is cubic 3C form of SiC. This assumption has not been well established experimentally. All 3C films investigated so far were highly defective with high densities of stacking faults, dislocations, and double positioning boundaries. It is not clear if the stacking faults formed as the result of post growth deformation or are the consequence of fundamental polytype instability.

The definitive answer was obtained in this study by the transmission electron microscopy analysis of defect-free mesas. An example of the TEM composite cross-sectional image is shown in Fig. 3. The mesa etched in the 4H substrate is marked in the lower portion of the image with the thin section of the 3C deposit is shown in the upper portion of the figure. Some of the 3C layer has been removed during thinning process using ion beam milling. The maximum thickness of the 3C layer on the right side of the figure is approximately 20 microns. Careful inspection of the entire cross-section revealed contrast associated with extended defects only at the interface between 4H and 3C. The more detailed analysis of these defects is presented in the next section. The volume of the epilayers is defect free. In particular, we have examined this and two other mesas for the presence of stacking faults perpendicular to the growth direction and parallel to the basal plane of the substrate. If nucleated during growth, such faults would be expected to propagate across the entire mesa leaving no trace on the mesa surface. As a consequence, examination of the cross-section at any location would detect every fault present in the entire volume of the original mesa.

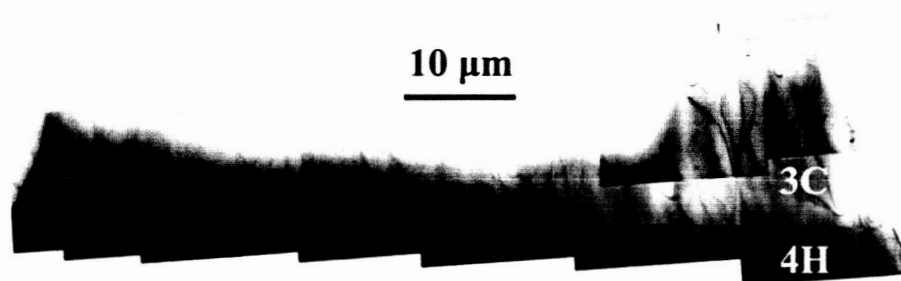


Fig. 3 Bright field TEM micrograph of the defect free mesa. The line contrast in the 3C film is due to thickness fringes. There are no extended defects visible in the entire 3C cross-sectional area.

Higher magnification of the right edge of the 3C mesa is shown in Fig. 4. The imaging conditions were selected in such a way as to produce a sharp contrast of the stacking faults (an example of faults in the defected mesa is shown in Fig. 5). It is evident that the



stacking sequence throughout the entire 20 microns of 3C growth in a defect-free mesa corresponds to perfect 3C cubic polytype.

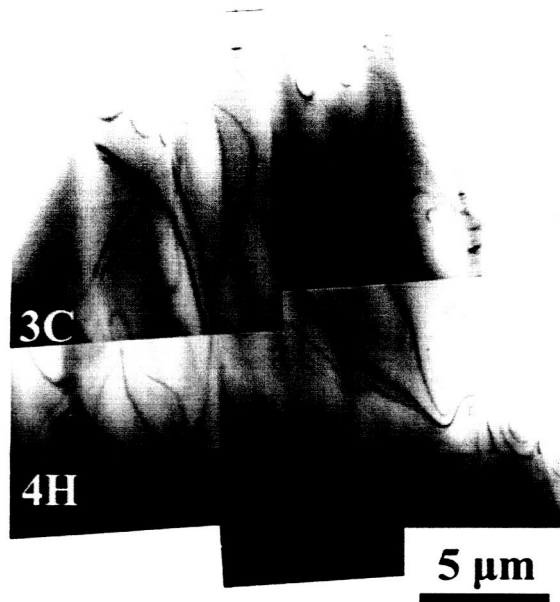


Fig. 4 Higher magnification image of the right edge of the mesa shown in Fig. 4

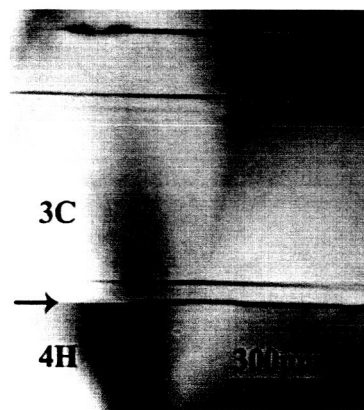


Fig. 5 TEM micrograph of the stacking faults in defected mesa. The 3C/4H interface is marked with an arrow. The line contrast in the 3C layer corresponds to stacking faults viewed edge-on.

### **Dislocations at the 3C/4H interface**

The a-lattice parameter of the 4H-SiC polytype is 3.073 nm while a corresponding value of 3C is 3.083 nm. The in-plane lattice mismatch between the two is  $3.3 \times 10^{-3}$ . During deposition of 3C epilayers on 4H substrate, the layer is expected to initially grow fully strained matching the lattice parameter of the substrate. Since the energy of the strained layer increases linearly with thickness while the energy of the misfit dislocation network that would lead to strain relaxation is constant, at some critical thickness the layer is expected to relax by introduction of dislocations. The value of critical thickness can be calculated by two methods proposed by People and Bean (People and Bean 1985) and Mathews and Blackledge (Mathews and Blackledge 1974). The two values are 1.1  $\mu\text{m}$  and 25 nm, respectively. It is expected, therefore, that the 3C epilayer investigated in this work should be relaxed.

Typically, the strain in semiconductor epilayers is relaxed by dislocations nucleating on the surface of the growing film and gliding toward the interface on (111) planes. The result of such mechanism is expected to be the two dimensional network of misfit dislocations with line directions along three equivalent  $\langle 110 \rangle$  directions in the (111) interface plane. Misfit dislocations should have a Burgers vector of the  $a/2\langle 110 \rangle$  type inclined to the interface and forming  $60^\circ$  with the line direction. The distance between dislocations corresponding to complete strain relaxation is 33 nm. Also, one of the typical consequences of the relaxation mechanism described above is high threading dislocation density in the epilayers. For most relaxed semiconductor structure, the threading dislocation density is in the  $10^7$ - $10^9 \text{ cm}^{-2}$  range. Threading dislocations are not a thermodynamic necessity. One can envision a perfectly relaxed epilayer with the dislocation network confined only to the interface. This could happen if the dislocation nucleation rate was very small and dislocation loops could glide unimpeded through the epilayer and terminate at the mesa edges. This is usually not the case with the misfit dislocation fragment length being only in the micron range. This is due to strong pinning of the glide motion by other dislocations.

Some of the features associated with the strain relaxation and described above have not been confirmed experimentally. The most striking effect is the absence of threading dislocations on the top of defect-free mesas. The optical micrograph shown in Fig. 1 was obtained on the layer that has been etched in molten KOH. Such procedure delineates intersection points of threading stacking faults and dislocations. Both are clearly visible on the defected mesas (Figs 1 and 6) in the form of dark points and lines. They are conspicuously absent on top of the defect-free mesa. The total estimated threading defect density in such structures is below  $5 \times 10^3 \text{ cm}^{-2}$  and several orders of magnitude below expected density of relaxed structure. At the same time, the high resolution x-ray diffraction results indicate that layers as thin as 1 micron are at least partially relaxed.

A direct evidence of relaxation was obtained through observation of misfit dislocations located at or close to the 3C-4H interface (Fig. 7).

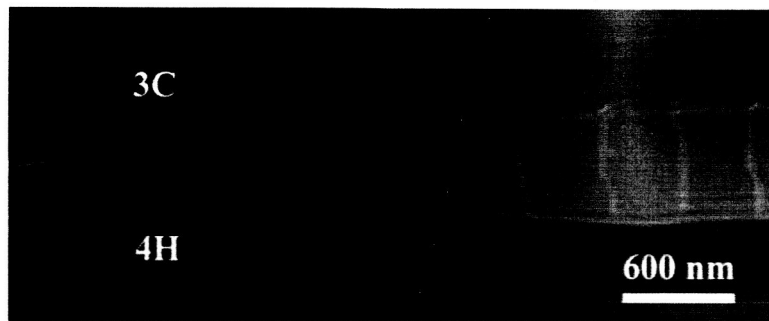


Fig. 6 TEM cross-sectional image of the 3C/4H interface. Almost vertical line contrast corresponds to dislocations at or near the interface.

The dislocation lines are either confined to the interface or very close to it with the line directions in all cases parallel to the interface. The dislocation lines are mostly parallel to each other and do not appear to form a network. The reason for this is under investigation. The distance between dislocations is about 300 nm what corresponds to 10% relaxation of the layer. This value is close to that reported by Dudley *et al.* (Dudley, Vetter *et al.* 2002)

Figures 7 and 8 present TEM images obtained in two beam conditions with the  $g$  vector either parallel or perpendicular to the interface (full analysis of the Burgers vector direction is impossible for this sample geometry). It is clear that the dislocation contrast with  $g=1-100$  is much stronger than for  $g=0004$ . This is a strong indication that the Burgers vector of this dislocation array is parallel to the interface. Although weaker, the contrast in Fig. 8 is not completely extinguished probably due to the mixed character of the dislocations.

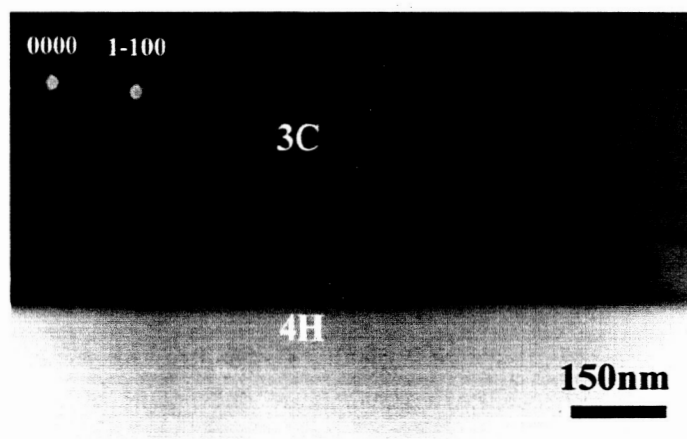


Fig. 7 Two beam dark field image ( $g=1-100$ ) of the dislocations at the 3C/4H interface

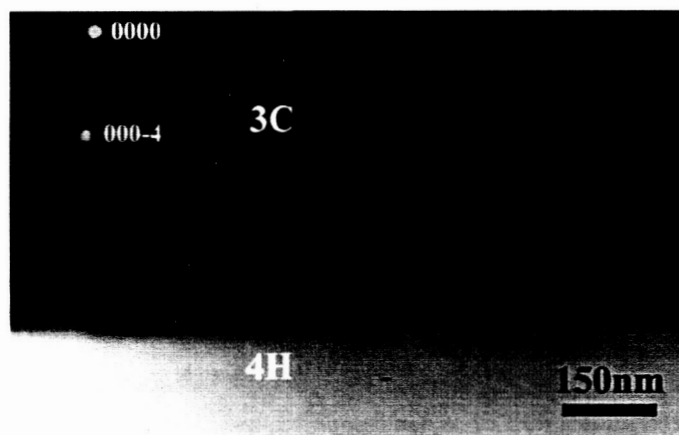


Fig. 8 Two beam dark field image ( $g=0004$ ) of the dislocations at the 3C/4H interface

Several features of the dislocation array shown in Figures 6, 7, and 8 are in disagreement with the relaxation model discussed earlier in the section. Specifically,

- (i) dislocations gliding from the top of the 3C layer toward the interface have to move in (111) glide planes and their trace at the interface has to be along  $\langle 110 \rangle$  directions.
- (ii) Burgers vectors of such dislocations have to be inclined to the interface plane in order to glide under the biaxial stress induced by the lattice mismatch.
- (iii) The threading segments of the misfit dislocations are not observed in densities typical of mismatched systems.

An alternative model of relaxation described below is in better agreement with experimental observations. Because of rather small size of the mesas the strain in the structure is not pure biaxial tension but is also expected to have a shear component in the plane parallel to the interface. Dislocations can nucleate not only on the top of the layer but on the sidewalls of the mesas as well. It is likely, therefore, that the arrays shown in TEM micrographs glided laterally along the interface. The salient features of this model are:

- (i) for the dislocations to glide in the plane of the interface their Burgers vector must be in the interface plane (in agreement with Fig. 7 and 8)
- (ii) the line direction of misfit dislocations does not have to be along any specific direction as dislocations can easily bend in the glide plane
- (iii) this relaxation mechanism would not introduce threading dislocation segments. In fact, none of the mesas, including defected ones, had the dislocation density approaching densities typical of hetero-epitaxial systems.

### **Dislocations in 4H substrate**

In addition to dislocations located at the 3C/4H interface, we have observed dislocation arrays in the 4H substrate close to the edges of the mesas (Fig. 9). In systems where thin lattice mismatched large area film is deposited on a much thicker substrate, the dislocations cannot be pushed into the substrate. The reason is much lower stress level

(approximately the stress ratio in the film and substrate are equal to one over the ratio of thicknesses). Moreover, the stress in the film is of opposite type than that of the substrate i.e. if the film is in tension then the substrate is under compression. The dislocations moving downward from the top of the film (under tension) will experience the upward force if they enter the substrate. The observation of dislocation arrays in the 4H substrate close to the interface is, therefore, an indication of qualitatively different stress state/relaxation mechanism.

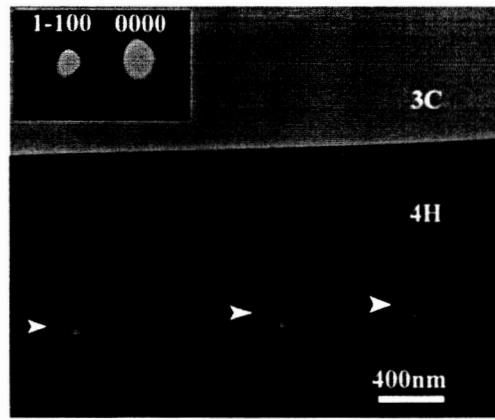


Fig. 9 Dark field image of the dislocations in 4H near the interface taken under the two beam condition ( $g=1-100$ ).

The dislocation array in question was observed close to the edge of the mesa. The dislocation lines are parallel to the interface lying in the basal plane of 4H polytype. The distance from the interface was about 1 micron with the dislocations spaced about 1 micron apart. The dislocations exhibited strong contrast in 1-100 reflection but were virtually extinguished for  $g=0004$  (Fig. 10). All of the above characteristics are in agreement with the new model of relaxation invoking glide along the interface plane.

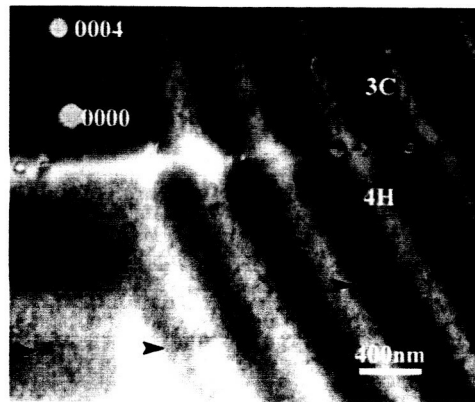


Fig. 10 Dislocation array in 4H substrate (two beam,  $g=0004$ ).

#### References:

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